

ATTORNEY DOCKET NO.: 05015.0209U3
APPLICATION SERIAL NO.: 09/997,455

Applicants respectfully submit that the present invention, as recited in claims 1-6, is novel over Wivell because the reference does not disclose, either expressly or inherently, each and every element recited in the claims. In particular, Wivell does not disclose a liquid continuous phase comprising a diol or that a silicone polymer is dispersed in a diol-containing continuous phase, as recited in claim 1 of the present invention. These arguments are more fully detailed below.

The emulsion compositions disclosed in Wivell are water-in-oil emulsions (col. 1, lines 14-15). As would be understood by those skilled in the art, a water-in-oil emulsion has an aqueous dispersed phase in an oil continuous (or external) phase. *See e.g.* the enclosed article from *Kirk-Othmer Concise Encyclopedia of Chemical Technology*, John Wiley & Sons, New York, 1985, pp. 415-417. Wivell discloses that the aqueous phase, *i.e.*, the dispersed phase of the emulsion, contains water, polyhydric alcohols with 2 to 6 hydroxyl groups, and a C₂-C₄ alkoxyate (col. 2, line 41, to col. 3, line 9). The oil phase of Wivell, *i.e.*, the continuous phase of the emulsion, contains volatile C₁₀-C₂₀ hydrocarbons and silicone oils (col. 3, lines 22, to col. 4, line 6). Wivell does not disclose that a diol may be present in the continuous phase. Nor does Wivell disclose that any silicone polymers may be dispersed in a diol-containing continuous phase. *See* Wivell, Examples 1-4, cols. 4-6.

Wivell discloses only silicone and volatile C₁₀-C₂₀ hydrocarbons in the continuous phase. In other words, the only diols disclosed in Wivell are found in the dispersed phase.¹ Therefore, because Wivell does not disclose any diol, much less 25-100 %, in the continuous phase, the present invention as recited in the claims is novel over this reference.

¹ Even though Wivell discloses that diols can be 1-60 % of the total composition (col. 2, line 54), the diols are, nevertheless, still in the dispersed phase because the Wivell composition is a water-in-oil emulsion.

Further, Wivell does not disclose silicone polymers dispersed in the continuous phase. To the contrary, the silicone polymers of Wivell actually make up the continuous phase and are therefore not dispersed within it. Thus, for this additional reason, amended claim 1 and those claims dependent thereon is novel over Wivell.

Moreover, the recitation of the transitional phrase "consisting essentially of" necessarily excludes materials that would change the basic and novel characteristics of the invention. That is, if the diol was in the dispersed phase in the present invention, the continuous phase would, of course, not contain diol. Since the claimed compositions comprise diol in the liquid continuous phase, it follows that the basic and novel characteristics would be changed by the inclusion of diols in the dispersed phase, as disclosed in Wivell.

Further, the present invention is not obvious in view of Wivell. Wivell discloses a continuous phase containing only silicone oil and C₁₀-C₂₀ hydrocarbons. Nowhere in Wivell is there a motivation, suggestion, or other teaching to prepare an emulsion with a diol in the continuous phase. Therefore, the present invention as recited in the claims is nonobvious over this reference.

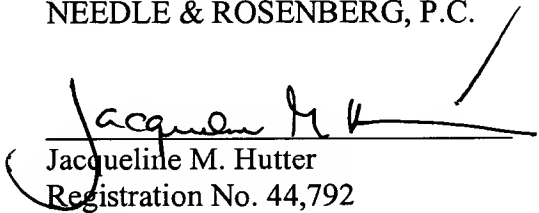
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CONCLUSION

In light of the above arguments and amendments the claims are believed to be allowable and Applicants respectfully request notification of same.

Payment in the amount of \$180.00 for the Information Disclosure Statement fee is enclosed. No further is believed due. However, the Commissioner is hereby authorized to charge any fees that may be required to Deposit Account No. 14-0629.

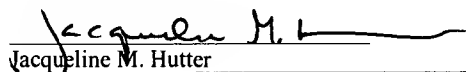
Respectfully submitted,
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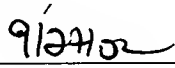

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VERSION WITH MARKINGS TO SHOW CHANGES MADE.

1. (Amended) A silicone polymer emulsion [comprising] consisting essentially of:
 - a. a silicone polymer;
 - b. a surfactant; and
 - c. a liquid continuous phase comprising a diol, wherein the diol comprises from about 25 to about 100% by weight of the continuous phase, and wherein the silicone polymer is dispersed in the continuous phase.

KIRK-OTHMER

**CONCISE ENCYCLOPEDIA
OF CHEMICAL TECHNOLOGY**

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interface with the metal positive with respect to the glass, many glass-metal systems form strong, hermetic seals. The experimental ranges cited above are typical. Any glass-metal system that can be sealed at all by the electrostatic method invariably has been found to be sealable at temperatures in the annealing range (ie, between the strain point and the annealing point). Seals are evaluated with regard to strength, hermeticity, and resistance to thermal shock.

Uses

Although there are broad applications for the electrostatic sealing method, only a few examples that demonstrate its benefits are given here. Since sealing temperatures are low and macro-distortion of the glass is minimal, the method is useful in the mounting of optical components. The application that has been investigated most widely is concerned with the mounting of silicon pressure transducers.

The electrostatic sealing process is commercially utilized by a number of companies under licensing agreements with Duracell Inc. which holds the applicable patents. As far as is known, one of the most popular applications is in the construction of pressure transducers.

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Westinghouse Electric Corp.

G. Wallis and D.I. Pomerantz, *J. Appl. Phys.* 40, 3946 (1969).
U.S. Pat. 3,397,278 (Aug. 13, 1968), D.I. Pomerantz (to P.R. Mallory & Co.).

EMBEDDING

The embedment of objects, the complete encasement of objects in a medium, practiced for centuries, has today become a science involving large numbers of scientists and engineers in all parts of the industrial world. The objectives of embedding may be either functional or decorative. The most important functional area is the embedding of electrical and electronic circuitry.

Embedding Process Considerations

Often, a given embedded product can be made by two or more of the various embedding processes such as casting, molding, potting, impregnating, or transfer molding. Thus, an analysis of the important comparative advantages of the methods is required. This section presents some of the most important of these considerations.

Primary embedding materials. Although waxes and bitumens are still used occasionally, most materials used for embedding are plastics. Liquid or easily liquefied plastics are most commonly used. As a class plastics are insulators. They therefore provide the necessary insulation required in electronic and electrical applications. Useful plastics fall into two large classes, thermoplastics and thermosets. Two properties to consider when choosing a plastic include viscosity and exothermic properties.

Transfer-molding resins. The most widely used materials for transfer molding are epoxies, although low pressure molding materials have been developed from silicones, diallyl phthalates, phenolics, and alkyds (see Alkyd resins), the first two enjoying wide usage.

Thermosetting Embedding Resins and Filled Compounds

Many chemically distinct embedding resins are available, and there are many variations in each group. They include epoxies, silicones, polyesters, polyurethanes, polysulfides, polybutadienes, low density foams, allylic resins, most often incorporating fillers.

Waxes and Thermoplastic Embedding Materials

Owing to the low temperature stability of waxes and bitumens, these materials have only limited embedding applications. The use of most thermoplastic resins is also restricted by the high molding pressures and temperatures required in their processing.

Decorative Embedding

The vast majority of embedding applications serve electrical or mechanical functional objectives, but decorative embedding also enjoys widespread use. Examples are primarily decorative items embedded in an unfilled resin having some degree of transparency, although nontransparent art objects are also cast using materials similar to those used in functional embedding. Also, scientific specimens are often embedded for cross-sectioning investigations.

Standards and Controls for Embedding

The importance of proper controls cannot be stressed too strongly from the standpoint of higher yield, lower cost, and increased efficiency, and from the standpoint of safety. Frequently, the potential hazard of handling embedding resins is not nearly so well recognized in an electronics plant as it would be in a chemical plant. Quality control should cover both manufacturing and production control. Finally, the key to reliable embedding techniques lies in material selection and maintaining the material quality by extensive testing.

C.A. Harper, *Handbook of Plastics and Elastomers*, McGraw-Hill Book Co., Inc., New York, 1975.

C.A. Harper, *Handbook of Materials and Processes for Electronics*, McGraw-Hill Book Co., Inc., New York, 1973.

H. Lee and K. Neville, *Epoxy Resins*, McGraw-Hill Book Co., Inc., New York, 1957.

EMULSIONS

An emulsion is a mixture of two or more immiscible liquids, one being present in the other in the form of droplets. Industrial emulsions frequently contain a solid as a third ingredient. Strictly speaking, a mixture of a solid dispersed in a liquid is a suspension. A variation in which molten waxes are emulsified is called a wax emulsion, although it is in fact a suspension or dispersion at room temperature. In the classic emulsion, the oil may either be dispersed in the water (oil-in-water or o/w emulsion) or the water dispersed in the oil (water-in-oil, w/o, or inverse emulsion). This terminology is important because of the external phase, a key factor in emulsion formulation and design.

Emulsions are found in nature, two of the principal examples being milk and rubber latex. These emulsions are stabilized by natural emulsifying agents. In a like fashion, commercial emulsions require emulsifying agents.

Emulsions are used in a variety of fields such as textiles, leather, and metal treatment; foods, cosmetics, pharmaceuticals, and paints; in agricultural chemicals, polymerizations, cleaning, and polishing; and ore and petroleum recovery.

Emulsions are inherently unstable systems and the risk of deteriorating during storage is greater than with a nonemulsified product. Emulsion technology, though seemingly based on simple interface principles, is highly complex, especially when dynamic and static conditions are considered (see Cosmetics; Food processing; Textiles; Leather; Pharmaceuticals; Latex technology; Paint; Polymers; Polishes, Petroleum).

Properties

Emulsion properties and characteristics are not necessarily related to the properties of the main active or key ingredients. The properties can usually be tailor-made to suit various use/applications requirements. They are built into an emulsion during formulation. The physical properties of an emulsion depend chiefly upon the properties of the external phase; the phase-volume relationship, and the particle size and they are under a surprising degree of control. The properties of emulsions and the related controlling features are shown in Table 1.

Table 1. Properties of Emulsions

Property	Related controlling features
appearance	
clarity	
clear	small particle size; matched refractive indexes
translucent	medium particle size
opaque	large particle size; unmatched refractive indexes
color	
white	large particle size; unmatched refractive indexes
gray	medium-small particle size; unmatched refractive indexes
colors	colors in continuous phase
viscosity	
thick (high)	HIP ^a emulsion; small particle size,
thickeners in outside phase	
thin	LIP ^b emulsion with no thickener
dispersibility	
in water	o/w
in oil	w/o
ease of preparation	
high, easy	emulsifier, solution level; low viscosity concentrate
low, difficulties	low emulsifier level
re-emulsification	emulsifier selection; emulsifier level
stability	
high, good	emulsifier selection; emulsifier moderately high
low, poor	low emulsifier levels; emulsifier selected for other property
stable to electrolytes	emulsifier selection
on evaporation (o/w)	emulsifier selection; emulsifier level
spoilage	preservative selection; sterile packaging
wetting-spreading	
high	emulsion type; emulsifier selection
low	emulsifier selection
particle size	
small	emulsifier selection; emulsifier level
large	emulsifier level

^aHIP = high internal phase.^bLIP = low internal phase.

Type of emulsion. The choice of the phase that is external or continuous is defined by the type of emulsion: o/w or w/o. In general, o/w emulsions conduct electricity, are dilutable with water, feel like water, dry (lose water by evaporation), are corrosive in comparison with w/o emulsions, and in general, exhibit properties expected of an aqueous liquid. On the other hand, w/o emulsions conduct electricity poorly, if at all, are usually diluted with oils or solvents rather than with water, feel more like oil, resist drying or loss of water although they will lose a volatile solvent readily if present in the continuous phase, are difficult to wash away, are less corrosive than o/w emulsions, and, in general, exhibit the properties of the continuous oil phase. There are also, in very few instances, dual emulsions, in which some water is dispersed in oil which is in turn dispersed in water, or the reverse. They exhibit properties of the external phase.

Volume ratio of external to internal phase. A low internal phase emulsion assumes the overall characteristics of the external phase. On the other hand, high internal phase emulsions exhibit higher apparent viscosities as the internal phase volume ratio increases. This effect is not pronounced until the internal phase volume exceeds 50–60% of the total volume, then the apparent viscosity increases rapidly and can exhibit characteristics of a gel or paste.

Particle size. Table 2 shows the effect of particle size on the appearance of the emulsion. The same range of particle sizes is of interest in formulating for stability; the smaller the particle size, the greater the

stability. However, stable emulsions can be formulated with particles as large as 1–2 μm . Since a smaller particle size emulsion is usually more costly to manufacture, formulation is usually a compromise of application needs/cost. The particle size of an emulsion may be reduced by increasing the amount of emulsifier, improving the selection of emulsifier with respect to both HLB (hydrophile-lipophile balance) and chemical type, modification of the method of preparation of the emulsion, ie, by the use of phase inversion to provide an extended internal phase at the time of inversion to the final emulsion type, and improved and/or increased energy input by changing or enhancing the agitation.

Formulation

In emulsion formulation, the goal is to achieve the best combination of emulsion properties to fulfill the application needs and stability requirements. The key or active ingredient(s) may or may not be the major ingredient. The main ingredient is most frequently the continuous phase and this dictates the type of emulsion, o/w or w/o, a pivotal decision. For economic as well as technical reasons, most commercial emulsions are o/w and have low oil (internal) phase levels. If a higher viscosity is desired with an emulsion of this type, a suitable soap (gel) may be incorporated in the emulsifier system or thickeners (gums, starches, proteins, and polymers) may be added. Moderate amounts of emulsifier(s) are usually used, ranging from 5–20 wt% of the oil/wax mixture. The level of energy input during manufacturing will influence the level of emulsifier. The emulsifier blend is best if soluble or dispersible in the oil/wax. Coupling with alcohols, glycol polyethers, fatty acids, traces of water, etc., are desirable. The emulsifier should be chosen to give the best balance of emulsification ease and emulsion stability; these characteristics do not always occur at the same HLB or with the same chemical type. If the product is to be an emulsifiable concentrate, it must be formulated to be self-emulsifiable. Some products may require deposition via controlled instability or a chosen particle charge that renders the particles substantive.

Choice of emulsifier. The use of the least amount of emulsifier is possible when a choice is made that most nearly matches the requirements for ionic type, HLB, and emulsifier chemical type. The choice of ionic type—anionic, cationic, amphoteric, or nonionic—will influence many properties of the final emulsion. The HLB is an expression of the relative simultaneous attraction of an emulsifier for water and oil (or for the two phases of the emulsion system being considered). It is determined by the chemical composition and the extent of ionization of the emulsifier. For example, ionic emulsifiers change HLB values radically with change in pH and/or salt content of the formula whereas nonionic emulsifiers exhibit a more constant HLB under these circumstances. The HLB value of commercial emulsifiers is usually available from their suppliers. It can be approximated by observing the dispersibility of the emulsifier in water, Table 3. It is usually best to achieve the desired HLB by blending a low HLB emulsifier with a high HLB emulsifier, both being of the same chemical family. An indication of required HLB values for many petroleum oils and waxes is shown in Table 4. Various oils have differing required HLB values, especially for differing applications, but even with these rough guidelines testing of a selected variety of blends of emulsifiers can cover a range of HLB and chemical types that will allow selection of the best emulsifier. Emulsifier chemical type is a more elusive match, though trial of surfactants, eg, laurate, stearate, oleate, etc, at the same HLB blended value will allow correlation of chemical type with stability and other properties.

Table 2. Effect of Particle Size of Dispersed Phase on Emulsion Appearance

Particle size	Appearance
macroglobules	two phases may be distinguished
greater than 1 μm	milky-white emulsion
1–0.1 μm	blue-white emulsion, especially a thin layer
0.1–0.05 μm	gray semitransparent, dries bright
0.05 μm and smaller	transparent, dries bright

Table 3. HLB of Emulsifiers by Dispersibility in Water

Dispersion characteristics	HLB range
no dispersibility in water	1-4
poor dispersion	3-6
milky dispersion after vigorous agitation	6-8
stable milky dispersion	8-10
translucent to clear dispersion	10-13
clear solution	13 +

Table 4. Emulsion Properties vs HLB or Required HLB Values for Hydrocarbon Oils / Waxes

Required HLB	Type of emulsion	Particle size, μm
4 to 8	w/o	0.5 to 3
9 to 12	o/w	0.1 to 3
13 to 14	o/w, detergency	
15 to 18	o/w, solubilization	less than 0.1

Emulsion testing. During formulation studies, the test emulsions should be evaluated not only for ease of emulsification but also for the important desired application properties and for stability to various temperatures including freeze-thaw conditions, and to agitation as anticipated in shipping. It is important to note that stability for commercial purposes may occur by either creaming or by coalescence of the internal phase. The latter is far more serious with respect to final use unless the product is re-emulsifiable.

Manufacturing procedures. In any formulation study, a tentative manufacturing procedure, including types of equipment and order of addition of ingredients, should be selected based upon equipment costs and availability. Power input cost must be balanced versus emulsifier costs. For example, an emulsion prepared on a colloid mill may require less than a tenth of the amount of emulsifier required if the emulsion were prepared in a tank with propeller agitation. On the contrary, the latter method can provide much smaller particle size. Order and rate of addition of ingredients can have a profound effect on the quality of the emulsion and must be tested in a variety of permutations and combinations. After a suitable choice is made, all further testing should follow the procedure unflinching. Generally, all the oils and waxes and other oil-soluble ingredients are combined as the oil phase. Polyols are combined with the water. However, salts are best added to o/w emulsions with the last part of the water, after inversion has occurred and a good primary emulsion has been established. In most instances of liquid-liquid emulsification, ambient temperature is preferred. Whenever elevated temperatures are used, the partition of the emulsifier between the oil and water may change, and modify the emulsification. With some equipment, having large heat input, some type of cooling must be effected.

Preservation. Since emulsions are often combinations of fatty materials and water and sometimes contain carbohydrates, they are good substrates for bacterial growth. Rarely are emulsions packaged under sterile conditions, hence preservatives are usually required (see Industrial antimicrobial agents). Some of the more common preservatives employed in emulsions include benzoic, propionic, and acetic acids and their salts and esters, also aldehydes and guanides.

Safety

Most emulsifiers are of low toxicity. Toxicity is mainly of concern in food and medicinal fields. These emulsions are subject to FDA scrutiny. Emulsions containing toxicants must be carefully formulated since they offer differing paths for entering the environment. Of course, emulsifiers can aid antipollution efforts by helping to clean up oil spills, etc., and aid in tank cleaning.

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ICI Americas Inc.

P. Becher, *Emulsions: Theory and Practice*, Reinhold Publishing Corp., New York, 1965.

P. Becher, ed., *Encyclopedia of Emulsion Technology*, Marcel Dekker, Inc., New York, 1979.

J.C. Johnson, "Emulsifiers and Emulsifying Techniques 1979," *Chem. Technol. Rev.* (125), 16 (1979).

ENAMELS, PORCELAIN OR VITREOUS

In the United States, the term porcelain enamel designates the glassy coating on metal; however, in some other countries, vitreous enamel is the more common term for the same glassy coating. The ASTM defines porcelain enamel as a substantially vitreous or glassy inorganic coating bonded to metal by fusion at a temperature above 425°C. Ceramic coatings, another term used for coatings on metal, connotes emphasis on the protective feature of the coating for the metal (see Refractory coatings). Ceramic coatings are often formulated and designed to contain mainly crystalline rather than glassy material (see also Colorants for ceramics; Glass).

Porcelain enameling protects against corrosion, decorates, and resists the attack of alkalis, acids, and other chemicals. This material is a nonporous sanitary coating imparting no odors or tastes. Since it is entirely inorganic, it does not serve as a feedstock for microorganisms. Because of its sanitary aspects, its protective and strengthening function, and its decorative character, porcelain enamel has been adopted as the most suitable material for bathtubs, laundry appliances, ranges, sinks, and refrigerator liners.

Typical physical properties include density 2.5-3.5 g/cm³; hardness 5-6 (Mohs scale); tensile strength 34-103 MPa (4,930-14,935 psi); compressive strength 1380-2760 MPa (200,100-1400,200 psi); modulus of elasticity 55-83 GPa (7,915,000-12,035,000 psi); and dielectric constant 5-10.

Classifications. Porcelain enamel is commonly classified by function, eg, ground coats, single frit, applied to ground-coated metal; by service, eg, acid resistant, alkali resistant, electrically insulating, thermal-shock resistant; by composition, eg, alkali borosilicate, titania, lead-bearing or leadless enamels; by metal coating, eg, sheet steel, cold-rolled steel; by decorative character, eg, clear, colored, stippled, mat; by opacifying material, eg, titania, zirconia, molybdenum oxide; by method of application, eg, wet process or dry process; by type of product, eg, appliances, jewelry, silos; and by firing temperature, eg, 540°C low temperature, 595-760°C.

The Enameling Process

The porcelain enameling process involves the re-fusing of powdered glass on the metal surface. The powdered glass is prepared by ball-milling a porcelain enamel glass engineered for specific properties. First the glass is smelted from raw batch materials. The enamel smelter is usually a box-shaped tank furnace. Continuous smelters, wherein the thoroughly mixed raw batch is fed in at one end and molten glass is flowing out at the other end, are common in commercial operations. Decomposition, gas evolution, and solution occur during smelting. After the molten glass has been smelted to a homogeneous liquid, it is poured in a thin stream of water or onto cooled metal rollers. This quenched glass, termed frit, is a friable material easily reduced to small particles by a ball-milling operation. Ball-milling the glass frit into small-sized particles can be carried out whether the frit is wet or dry (see Size reduction). Dry powders are used for dry-process cast-iron enameling and for electrostatic application on sheet steel (see Powder coating). Dry powders are also prepared and marketed for the subsequent preparation of slurries and slips used in the wet-process application techniques.

The electrostatic powder processing of porcelain enamel is similar to the electrostatic techniques used in preparing organic coatings (see Coating processes). Compared to the conventional wet-process porcelain enameling of sheet steel, the dry system eliminates the need for the clay suspending agents which represent refractory additions. As a consequence, these dry powders permit firing at a lower temperature as compared to that of the wet powders, thereby saving energy. At the